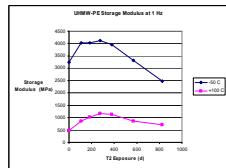
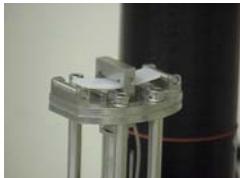


Tritium Effects on UHMW-PE, PTFE, and Vespel®



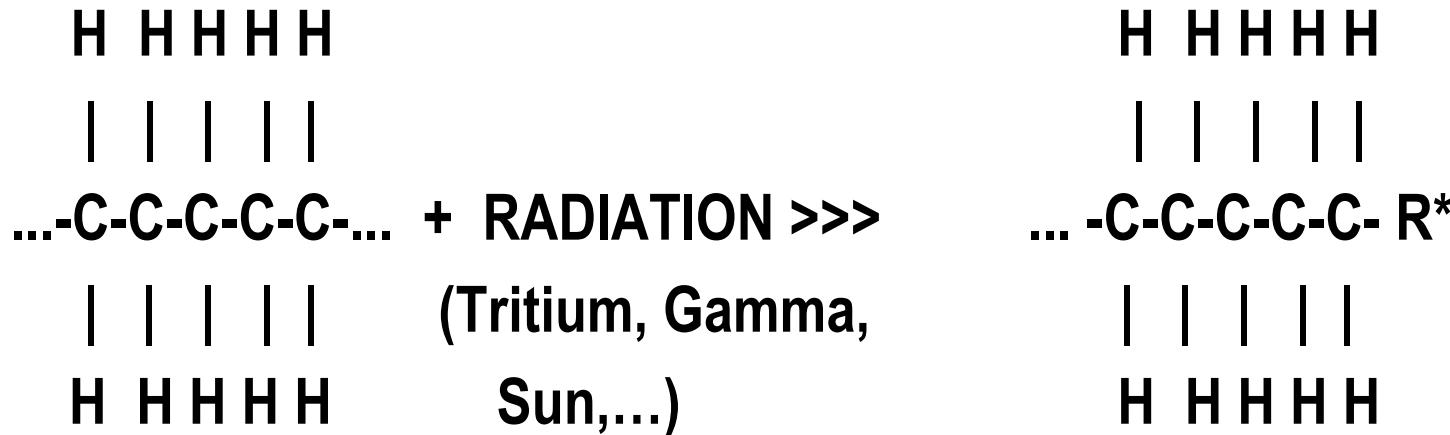
We Put Science To Work

Elliot A. Clark, Kirk L. Shanahan[#], Martin J. Pechersky

Materials Technology, [#]Hydrogen Technology

“Hydrogen Isotopes and Helium in Materials” Working Group Meeting
14 April 2005
Sandia National Laboratory, New Mexico

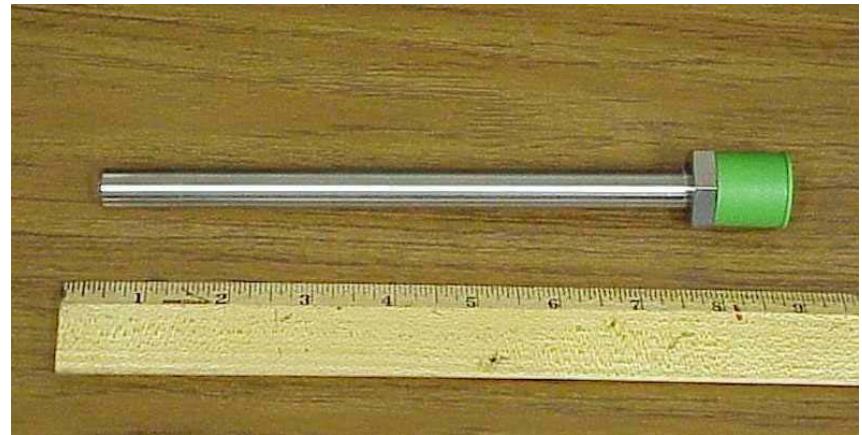
Radiation Damage of Polymers



- R* is a “Free Radical”, High Energy Temporary Chemical State
- R* Then Reacts: Chain Recombination, Cross-Linking, Scission vs. Bond Breaking
- Net Result: Either Stiffening (Hardening) or Degradation

Tritium Exposure & Characterization

- Materials: UHMW-PE, PTFE (eg. Teflon®), Vespel® polyimide
- Initially 1 atm Tritium, ambient temperature
- 3, 6, 9 ,12, 18, 24 Months Exposure Time
- Characterization
 - Dynamic Mechanical Analysis (DMA)
 - Dimension, Mass -> Density
 - Color Changes- Colorimetry
 - FT-IR
 - Change in Exposure Gas (Pressure, Composition)

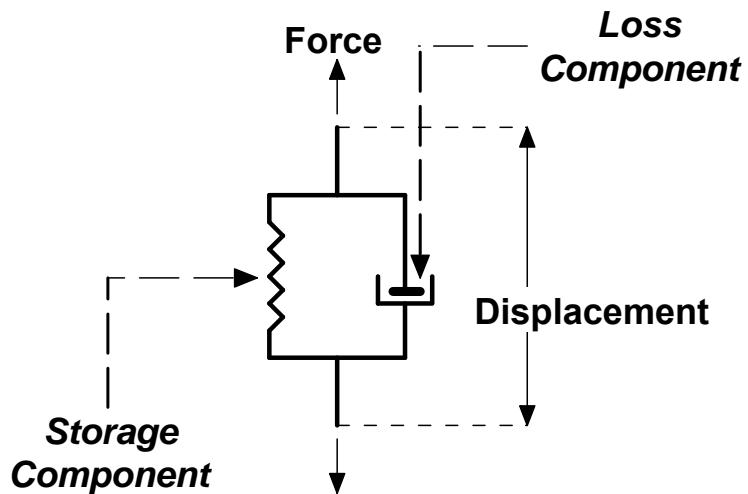


Modulus as Material Property

- Applied Force F, Sample Lengthens d:
 - Stress $\sigma = F/A$ (*A* is cross sectional area)
 - Strain $\varepsilon = d/L$ (*L* is original sample length)
 - Modulus K = σ/ε (*small strain*)
- Depending on Geometry, K is
 - Young's Modulus (Tensile) E
 - Shear Modulus G
 - Bulk Modulus B
 - ...

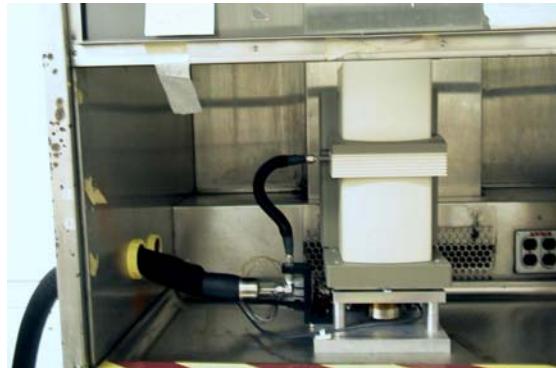
Elastic and Viscoelastic Deformation of Polymers

- Polymers have Instantaneous (Elastic) AND Time Dependent (Viscoelastic) Response to Load
- Complex Modulus $K^* = K' + iK''$ Describes This Behavior
 - Storage Modulus K' : Stored Elastic Energy in Polymer, Equivalent to Spring Constant
 - Loss Modulus K'' : Energy Dissipation in Polymer During Loading, Equivalent to Viscous Flow
- DMA Measures Force, Displacement and Phase Angle δ , Calculates K' , K'' . Multiple Frequencies, Temperature (Continuous ramp).

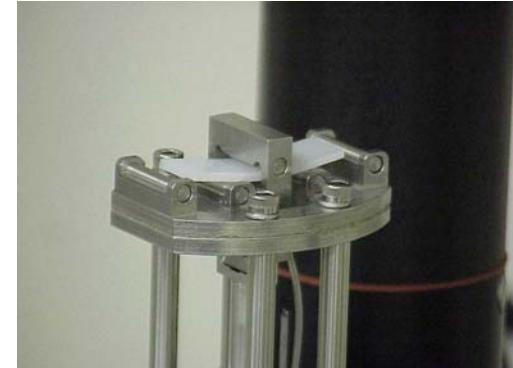


$$\tan \delta = \frac{K''}{K'}$$

Dynamic Mechanical Analysis



Mechanical Part in Tritium Hood

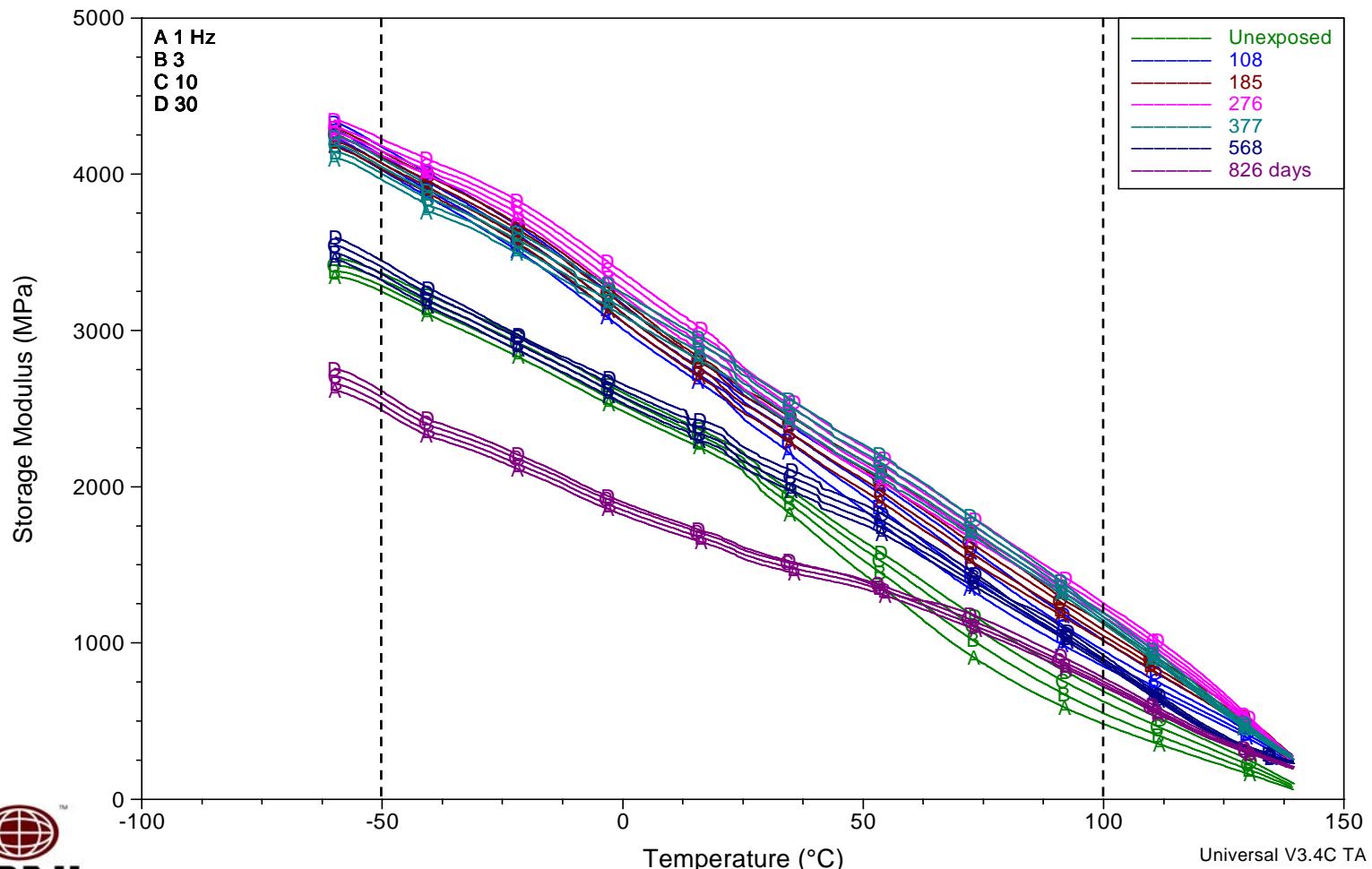


3 Point Bend Sample

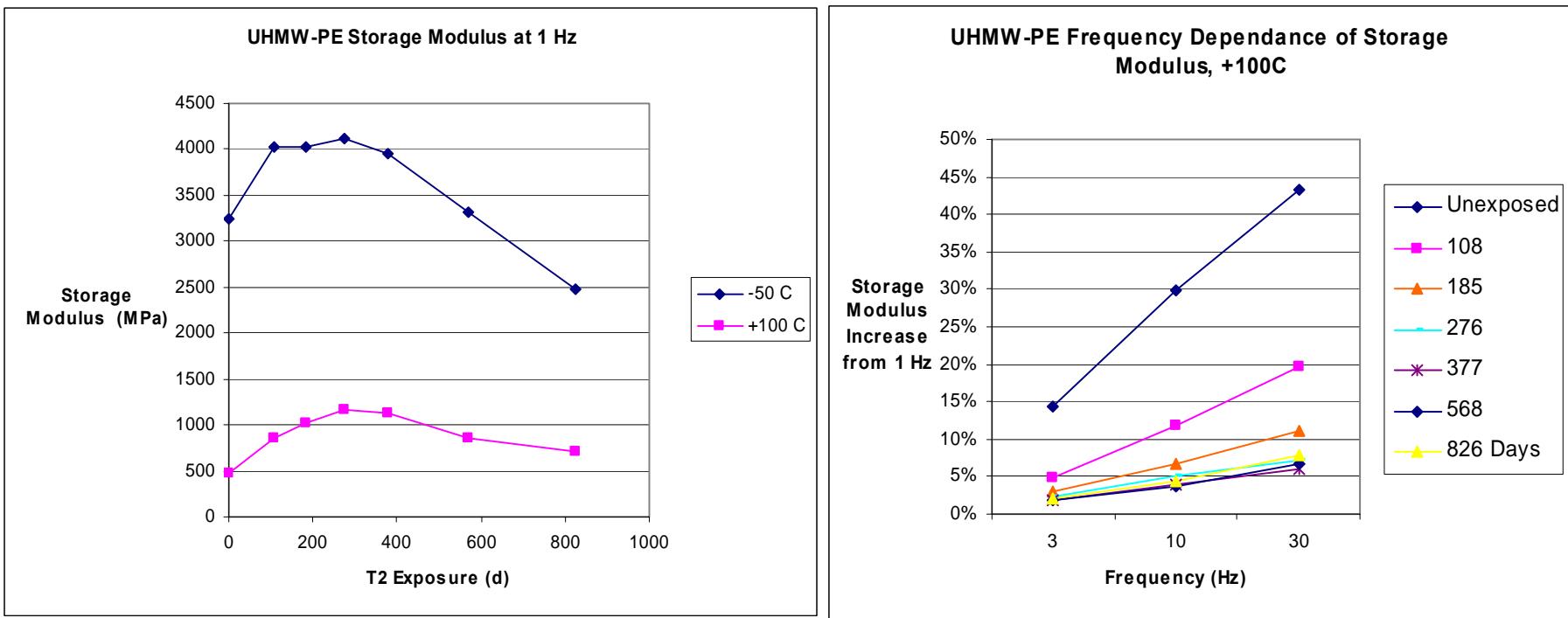
- TA Instruments 2980
 - Ramp 1°C/minute
 - -60°C to 140°C (UHMW-PE), 170°C (PTFE), 270°C (Vespel®)
 - Apply Sinusoidal Load at 1, 3, 10, 30 Hz Sequentially
 - TA Inst Engineered Mechanical Apart from Electronics, Base

UHMW-PE Storage Modulus

UHMW-PE exposed to 1 atm. T2 ambient temperature time in days

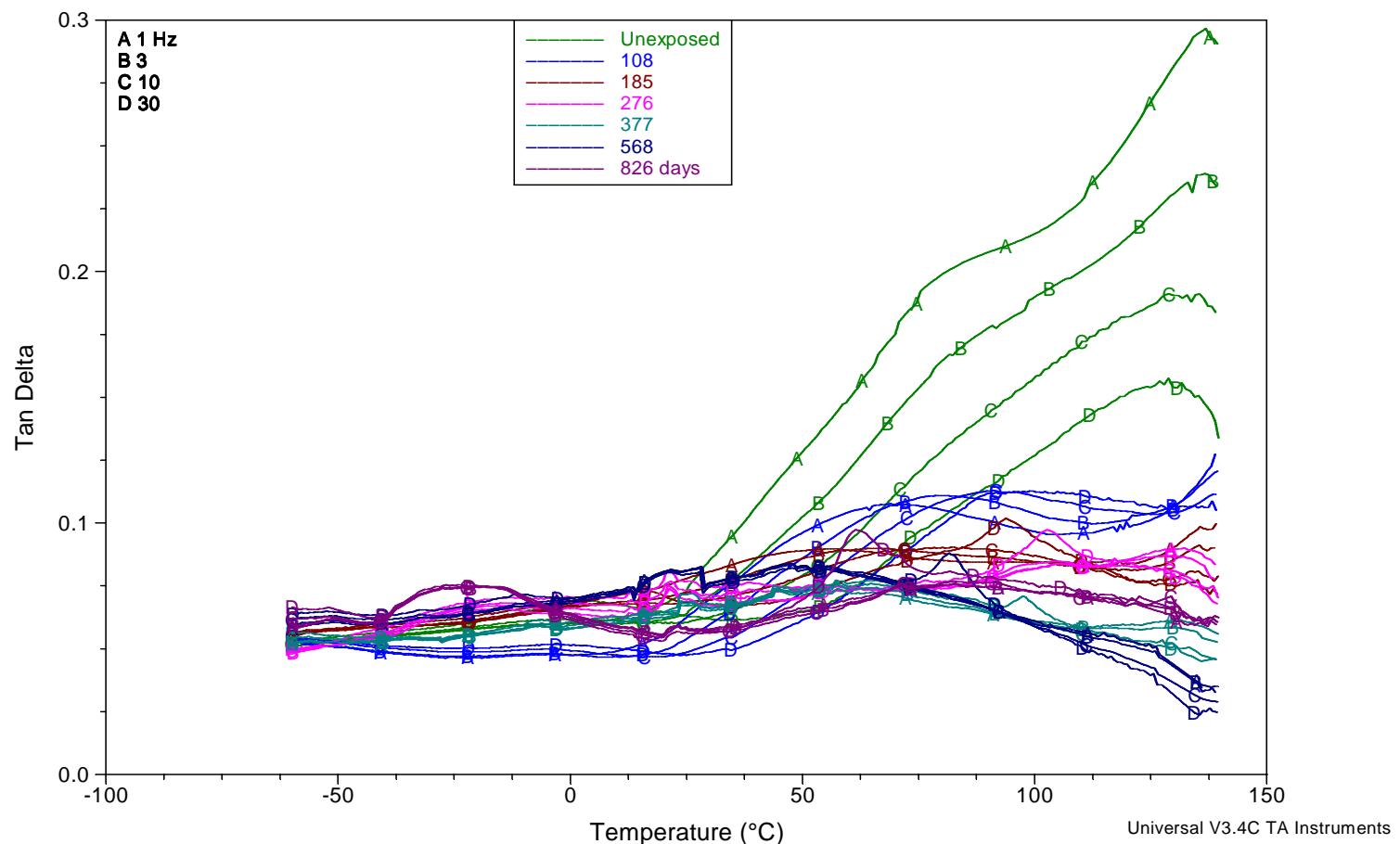


UHMW-PE Storage Modulus- Temperature and Frequency Dependence Changes with Exposure



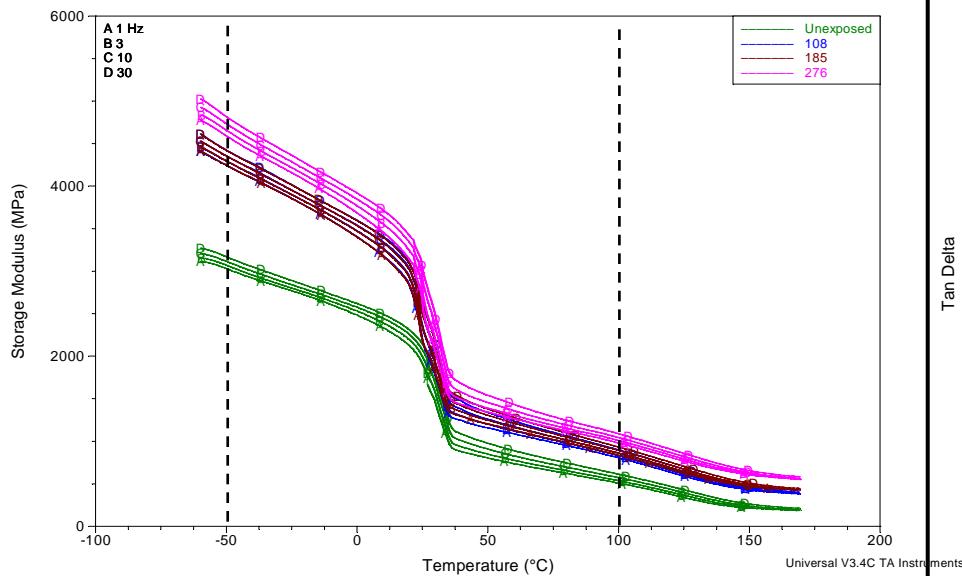
UHMW-PE: Tan δ

UHMW-PE exposed to 1 atm. T2 ambient temperature time in days

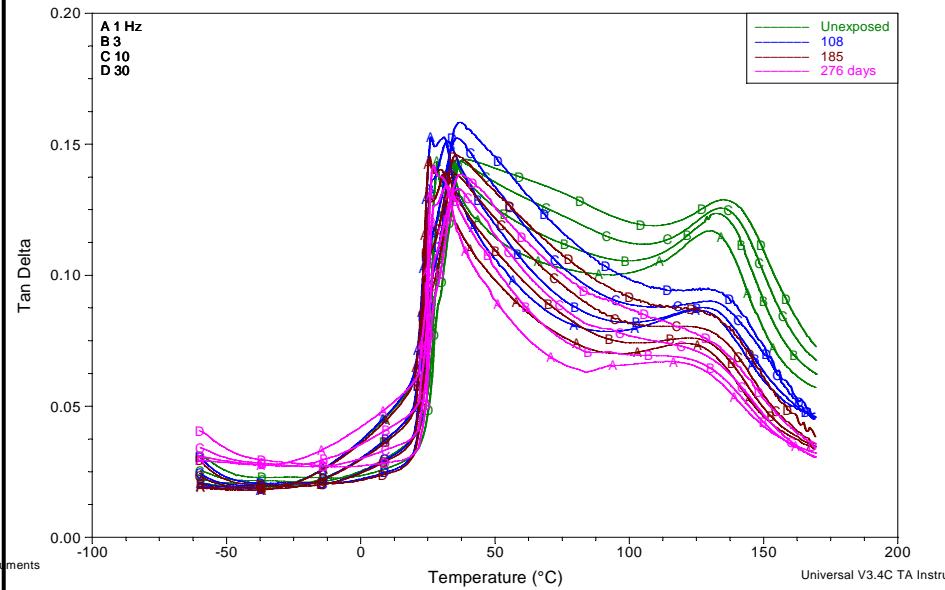


PTFE: Storage Modulus, Tan δ

PTFE exposed to 1 atm. T2 at ambient temperature



PTFE exposed to 1 atm. T2 ambient temperature

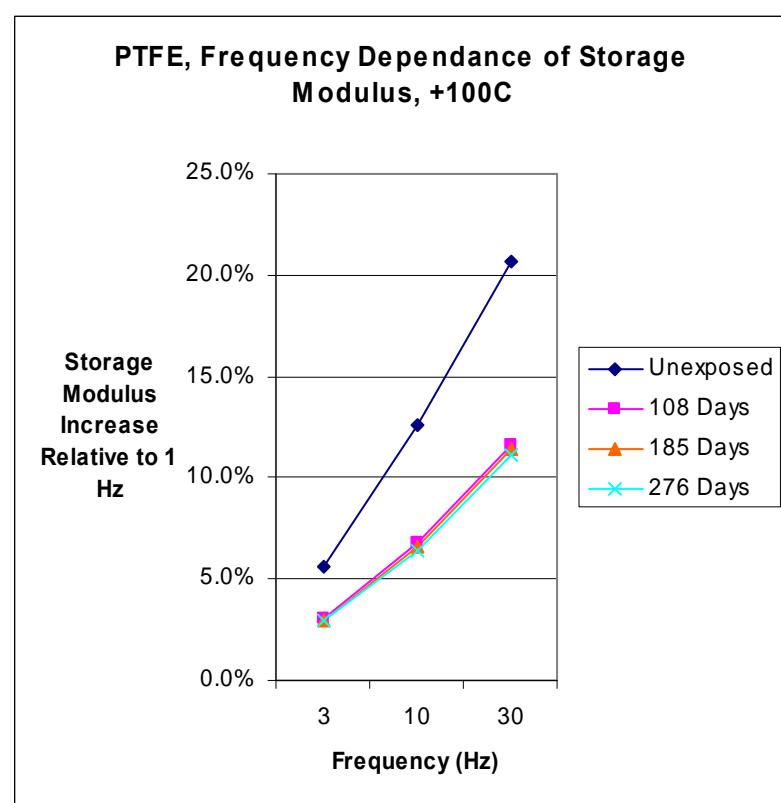
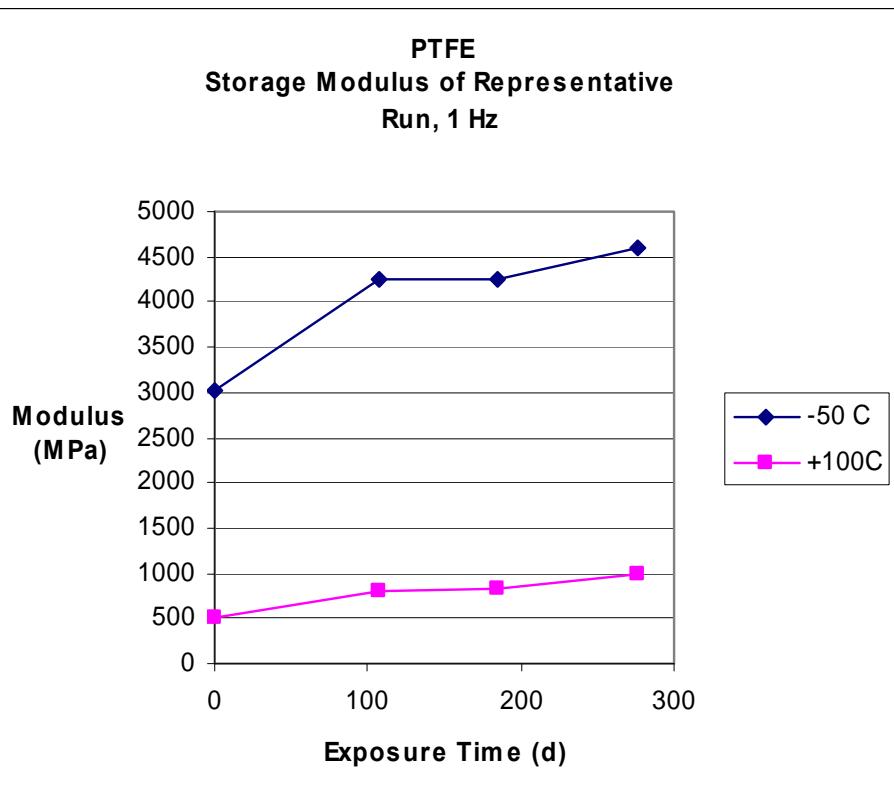


Storage Modulus

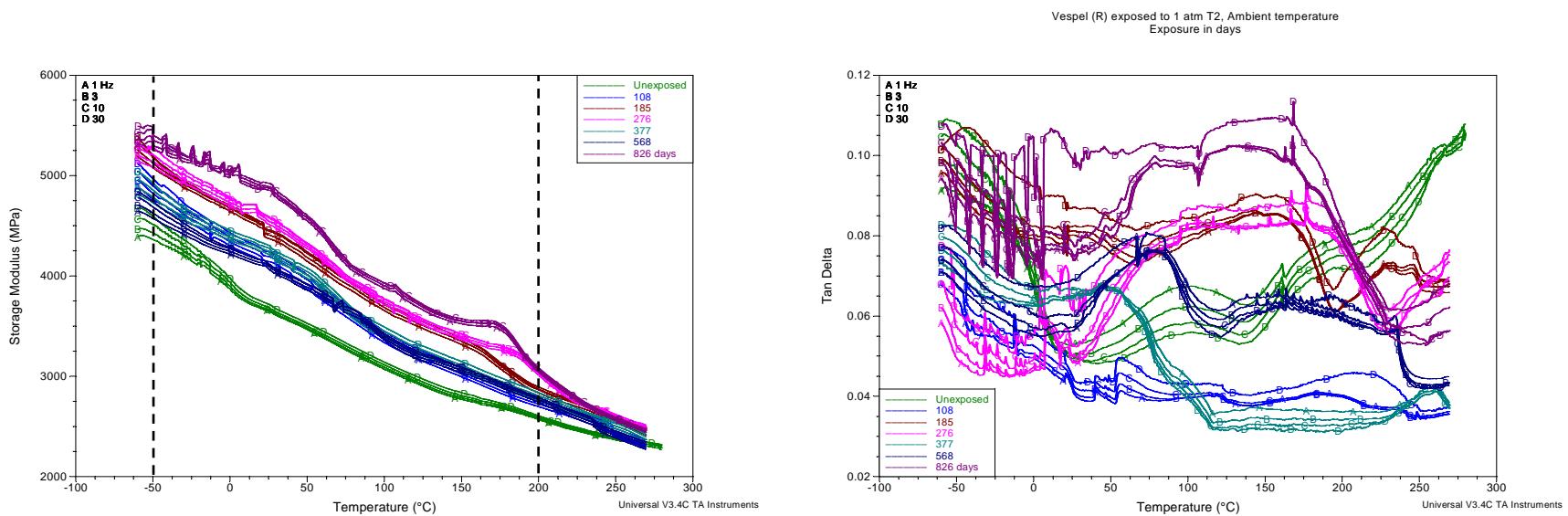
Tan δ

Samples Aged > 9 Months Broke When Handled
Too Weak to Test !

PTFE: Temperature and Frequency Dependence



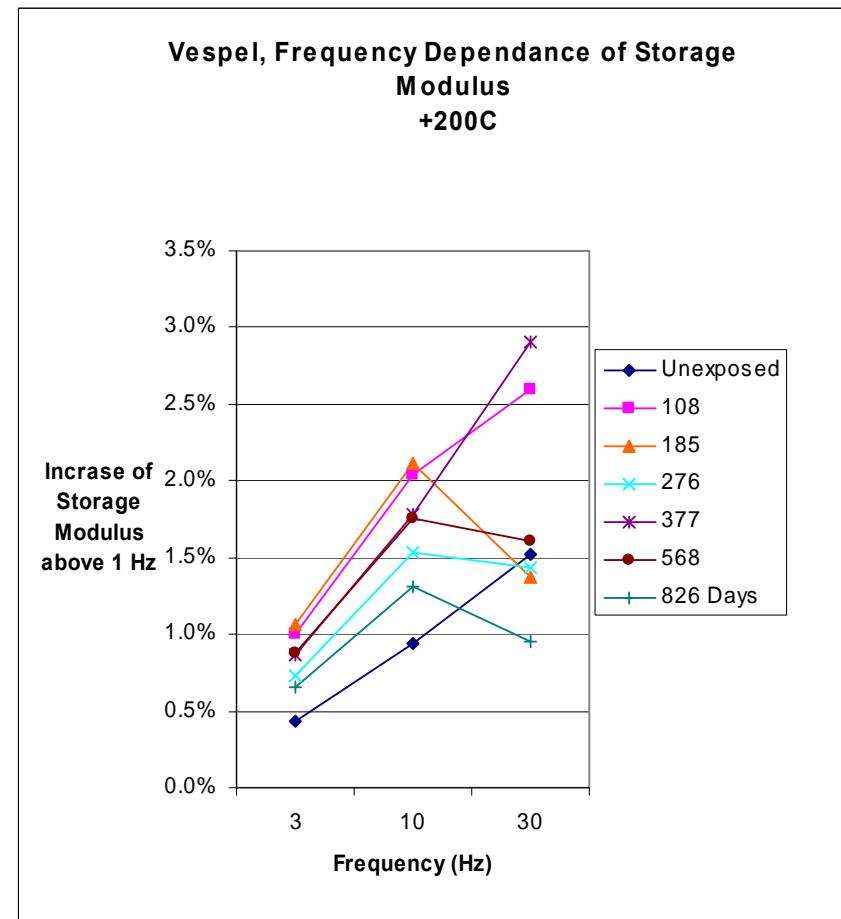
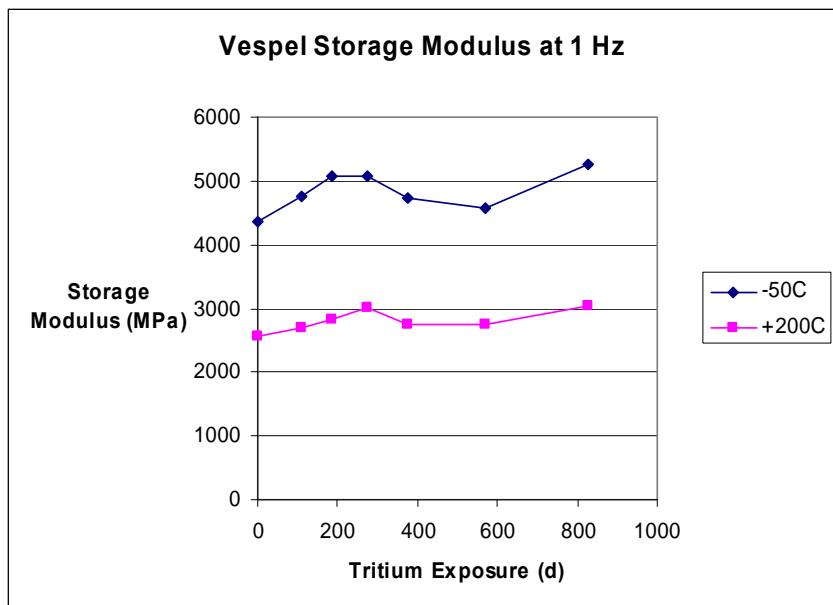
Vespel® polyimide: Storage Modulus, Tan δ



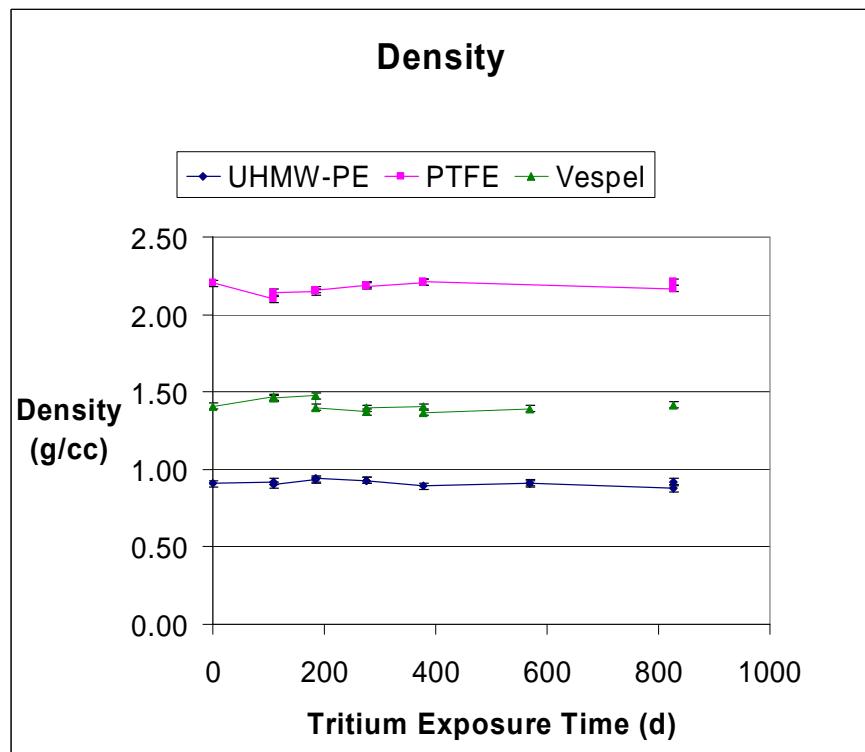
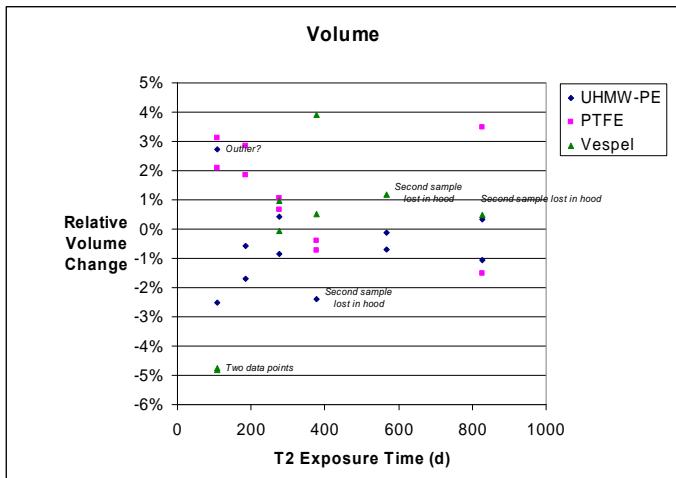
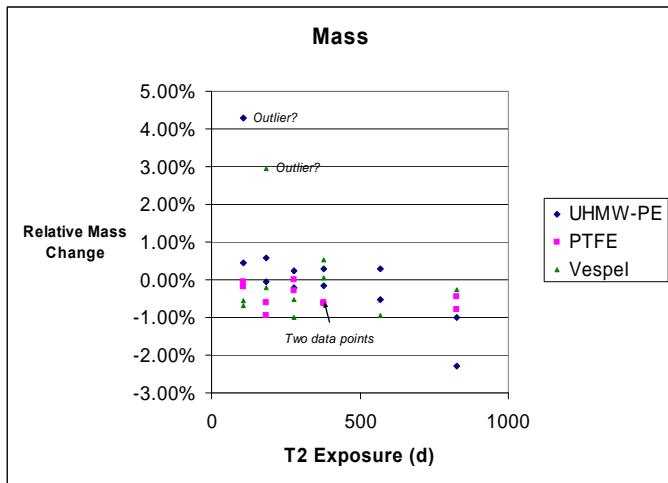
Storage Modulus

Tan δ

Vespel®: Temperature, Frequency Dependence

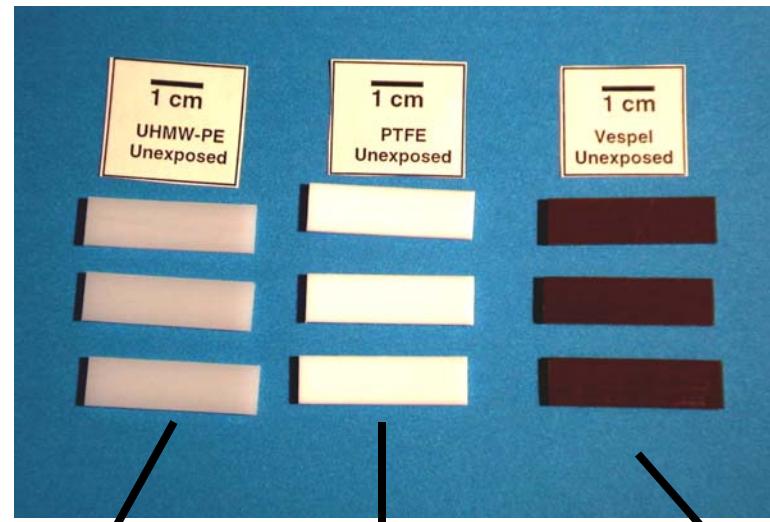


Sample Mass, Volume, Density

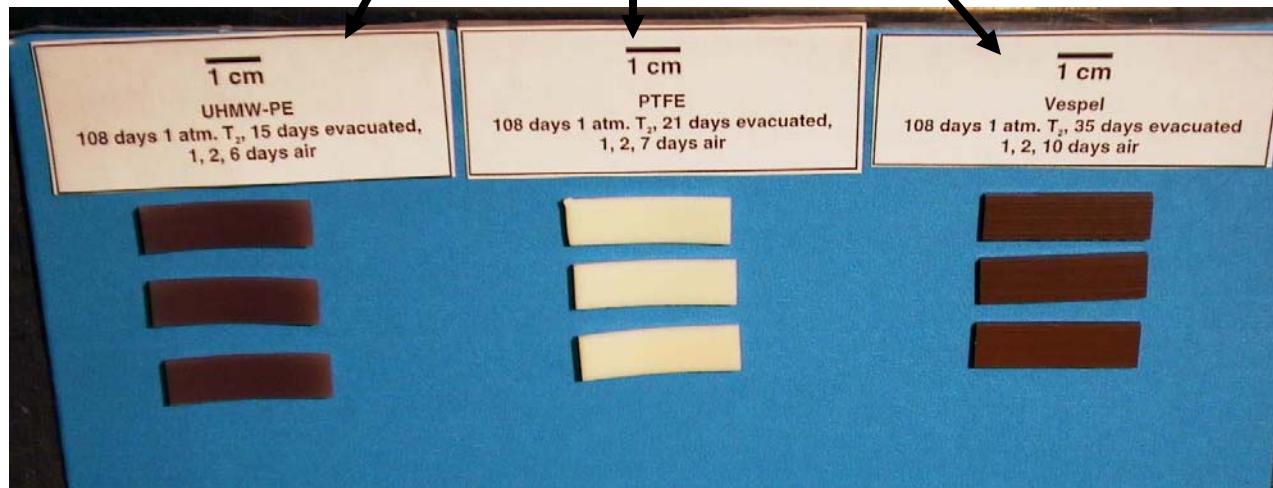


Color Change

Unexposed:



108 Days:

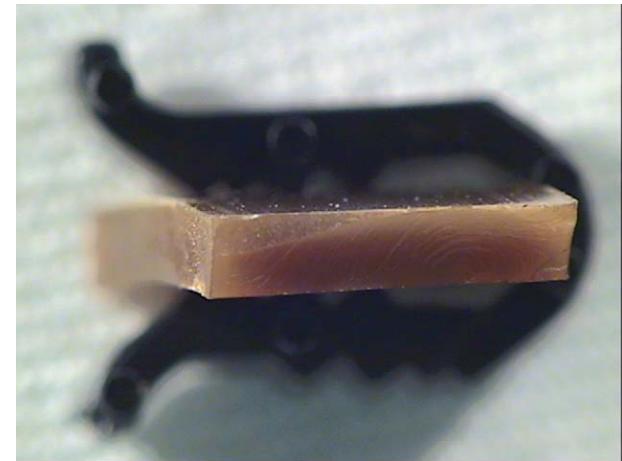


Color Change Differs Inside: 12 Month Exposure

PTFE



UHMW-PE

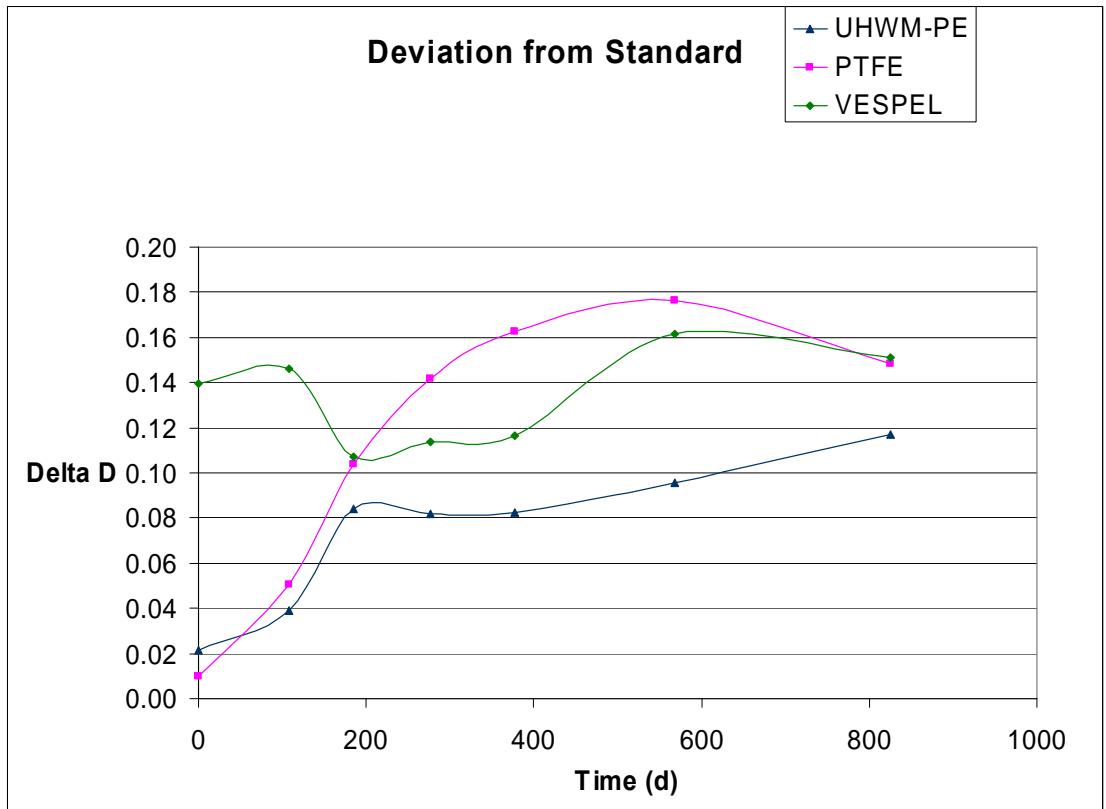


Colorimetry

CIE 1931 Chromaticity:
Color Expressed as x, y

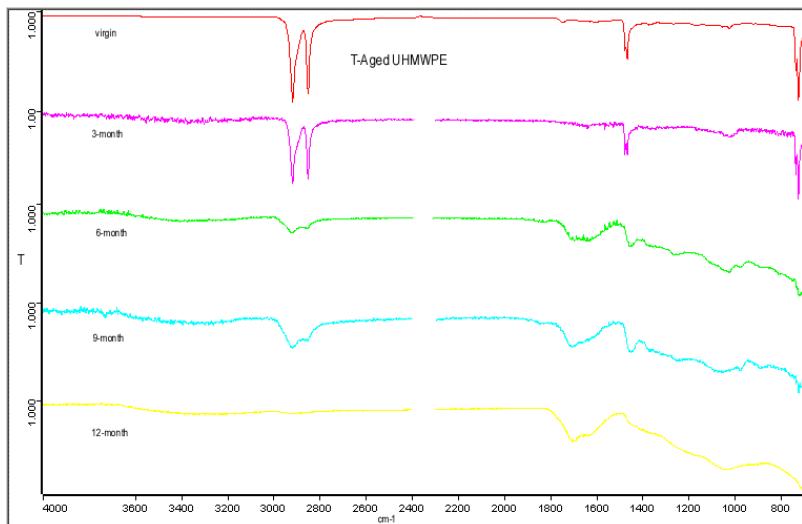
$$\Delta E = \sqrt{(x - x_{std})^2 + (y - y_{std})^2}$$

Color standard (white) x_{std} and y_{std}

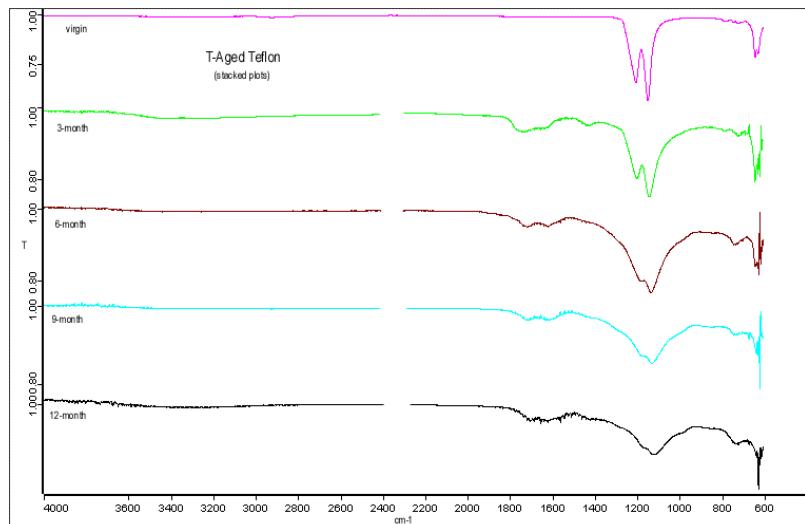


FT-IR Spectroscopy

UHMW-PE



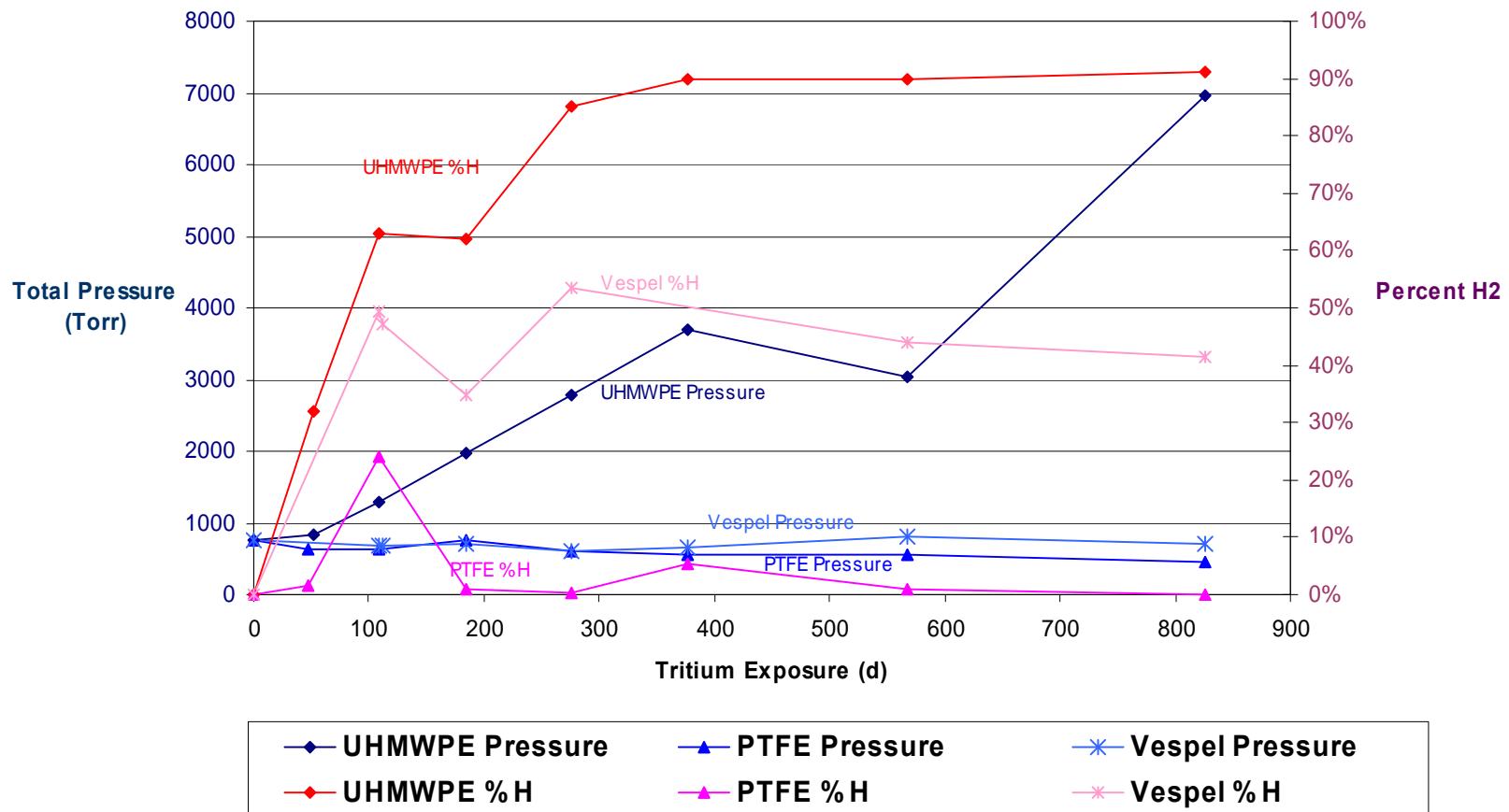
PTFE



Total Internal Reflectance- Results Uncertain (Instrument, Tritium Effects on Instrument)

Exposure Gas

Gas at End of Exposure



Summary

Exposure to 1 atm T₂ at Ambient Temperature for Up to 2.3 Years:

- UHMW-PE Significantly Affected (Storage Modulus, Tan δ, Internal and External Color, IR Spectroscopy). Significant Pressure Increase in Exposure Container (7000 Torr) and Isotope Exchange (90% H).
- PTFE Significantly Affected (Storage Modulus, External Color, IR Spectroscopy). Net Pressure Decrease in Exposure Container, Little Isotope Exchange (but some). *Samples Become Brittle > 9 Months Exposure.*
- Vespel® Much Less Affected- Small Increase in Storage Modulus Initially. Surprising Amount of Isotope Exchange in Exposure Gas (~50% H after 3 Months); Total Gas Pressure ~Constant.

Acknowledgments

- Kirk Shanahan: Sample Exposure, FT-IR, Gas Pressure/Composition
- Marty Pechersky: Colorimetry
- Wanda Britt: Sample Exposure, Tracking & Handling, Mass/Volume Measurements, DMA Operation
- David Bell: Sample Exposure
- Mike Thomas: DMA Installation